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TIME-DEPENDENT RESPONSE OF MI SiC/SiC COMPOSITES PART 1: STANDARD SAMPLES (PREPRINT)

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14. ABSTRACT
With the increased interest in using high performance ceramic matrix composites for advanced applications, long-term property behavior is of interest. In this work, time-dependent response of MI SiC/SiC composites (01/01 material) was experimentally evaluated under creep and dwell fatigue loading. A series of standard samples were tested at 815 degrees Celsius and 1204 degrees Celsius at various stress levels and multiple durations. All specimens showed primary and steady state creep responses. There were also some samples that showed tertiary creep response. Environmental degradation was empirically related to material response at different stress levels. Micrographic images of failed specimens revealed the existence of cavitations that were possibly caused by the creep strain at high stress areas.

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TIME-DEPENDENT RESPONSE OF MI SIC/SIC COMPOSITES PART 1: STANDARD SAMPLES

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ABSTRACT:

With the increased interest in using high performance ceramic matrix composites for advanced applications, long-term property behavior is of interest. In this work, time-dependent response of MI SiC/SiC composites (01/01 material) was experimentally evaluated under creep and dwell fatigue loading. A series of standard samples were tested at 815ºC and 1204ºC at various stress levels and multiple durations. All specimens showed primary and steady state creep responses. There were also some samples that showed tertiary creep response. Environmental degradation was empirically related to material response at different stress levels. Micrographic images of failed specimens revealed the existence of cavitations that were possibly caused by the creep strain at high stress areas.

INTRODUCTION

As Ceramic Matrix Composites (CMCs) are being considered for long duration applications, testing is needed to understand the material behavior under conditions of sustained load and temperature. Examples of long term applications can be found in ground base turbines for power generation where CMCs are being considered for combustor liners, turbine vanes and shroud applications [1,2]. These applications can see design times of up to 30,000 hours. Such long term applications are working to leverage the high temperature material capability while taking advantage of the weight reduction, reduced cooling and durability improvements that CMCs can provide over typical metals used in such applications.

As part of an effort to look into the long-term behavior of a CMC, a series of creep and dwell fatigue tests were undertaken. During the tests, strain was measured. The testing was done at 1204ºC with some testing done at 815ºC. The results of this testing will be shown and discussed.

PROCEDURE

Material Description

For this testing, the composite system interrogated was a Melt Infiltrated In-Situ BN SiC/SiC composite (MI SiC/SiC). This system has the most characterization done to date and is being considered for high temperature applications [3]. The MI SiC/SiC system has a stochiometric SiC (Sylramic™) fiber in a multiphase matrix of SiC deposited by chemical vapor deposition followed by slurry casting of SiC particulates with a final melt infiltration of Si metal. The specific MI SiC/SiC tested for this effort had 36% volume fraction fibers using a 5 HS weave at 20 EPI. The fibers are 10 μm diameter and there are 800 fibers per tow. This material system was developed by NASA-GRC and is sometimes referred to as the 01/01 material [4]. A cross section of this material is shown in Figure 1.

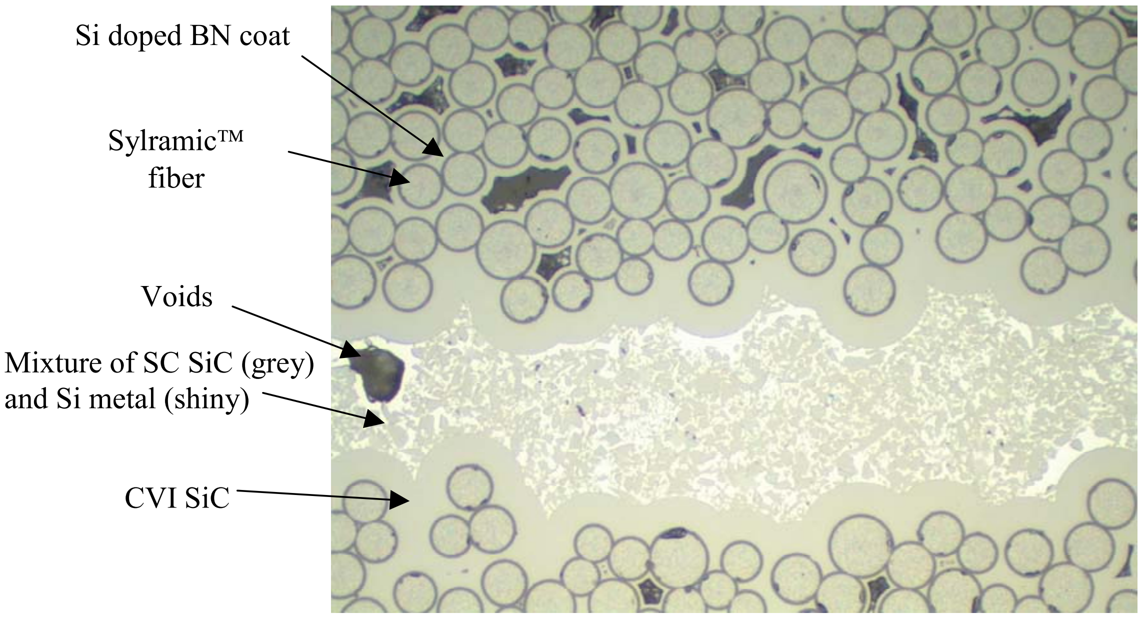


Figure 1. Cross section of Melt Infiltrated In-Situ BN SiC/SiC composite

Creep Testing

All creep testing was done in air using a SiC furnace. Temperature was controlled by thermocouples placed on the sample or by optical pyrometry. Load was applied using a dead weight using a lever arm. Strain was recorded using a 1” extensometer. Some of the testing allowed total strain to be recorded while other tests could only capture the creep strain. Testing was done at either 1204ºC or 815ºC.

Dwell Fatigue Testing

The dwell fatigue cycle for this effort was a creep type but the load was cycled off and on every 2 hours (R ratio of 0.1). All the dwell fatigue testing was done in air using a SiC furnace. Temperature was controlled by thermocouples placed on the sample. Load was applied using a dead weight with a lever arm. The lever arm is controlled by a cam system so that the load could be cycled off in a controlled manner every 2 hours. Strain was recorded using a 1” extensometer. One extensometer was used during the entire test so that the load and unload cycle was recorded and the modulus value could be calculated for every cycle. Testing was done at either 1204ºC or 815ºC.

Stress Level for Testing:

The vast majority of testing was done at stress levels of 110.4, 165.6 and 193.2 MPa. These values were picked based on the tensile curves at 1204ºC (See Figure 2.) The stress of 110.4 MPa is still in the elastic linear region of the material. The stresses of 165.6 and 193.2 MPa are in the knee of the stress-strain curve where some in-elastic strain is occurring indicating that the matrix is cracking allowing for some environmental attack into the material. Additionally, some limited testing was done at stress levels of 220.8 MPa and higher.

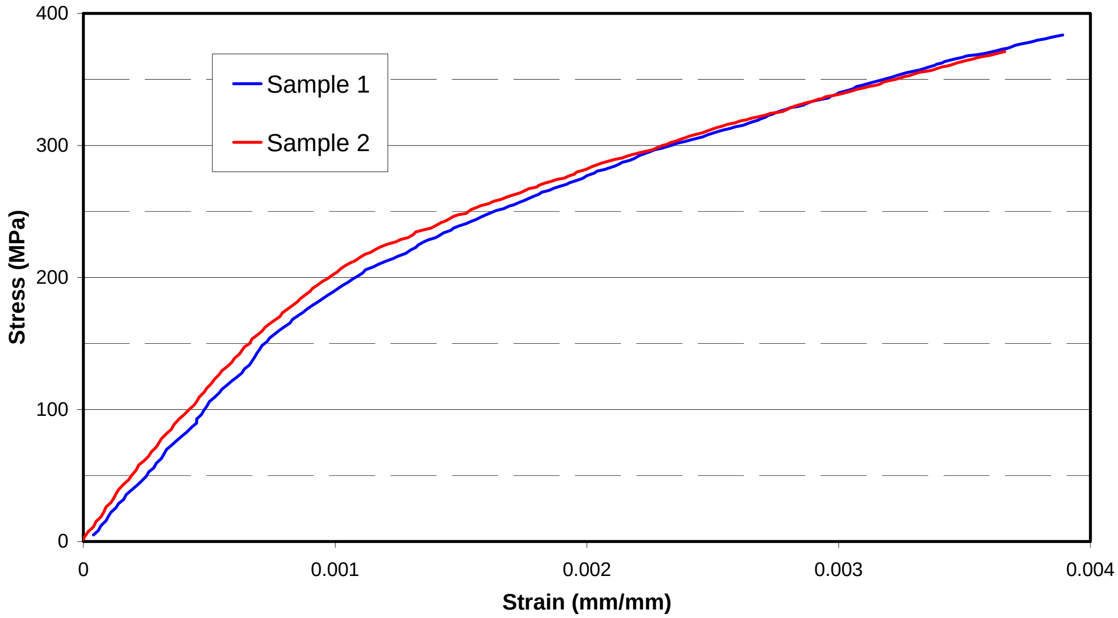


Figure 2. Stress-strain curves at 1204ºC for the MI SiC/SiC System being studied

RESULTS

Creep and Dwell Fatigue Testing Summary

The range of stresses and times that the material was exposed for under creep and dwell fatigue conditions are shown in Figure 3 where the range of stresses and exposure times are shown (without and with failure noted). All of this testing was done at 1204ºC. As can be seen, failures only occurred during creep testing when the material was exposed to high stresses or for long times. There was limited testing at 815ºC for durations of less than 250 hours and there were no sample failures.

Long Term Creep Testing Analysis:

As can be seen in Figure 3, there were only 8 samples tested for relatively long durations (>250 hours) at stress levels of 110.4 and 165.6 MPa. The resulting creep curves for this effort are shown in Figure 4. The curves for both the 110.4 and 165.6 MPa stress levels show both a primary and a steady state creep region. Some of the samples show tertiary creep. The steady state creep rate was determined for this testing after a review of the instantaneous creep versus time (see Figure 5). As can be seen in Figure 5 for these series of tests, the steady state creep region does not occur until near 250-300 hours. Hence, the steady state creep rate was determined from 250 hours on. At 110.4 MPa, the creep rate was found to be $3.4x10^{-10}$ while at 165.6 Mpa, the creep rate was found to be 6.3×10^{-10}. The creep rates for these conditions do not appear to be highly stress related since the exponents of the creep rates are the same and there is not a large difference in the magnitudes. In addition, this data is based on limited testing (2 samples for each condition) as shown in Figure 4.

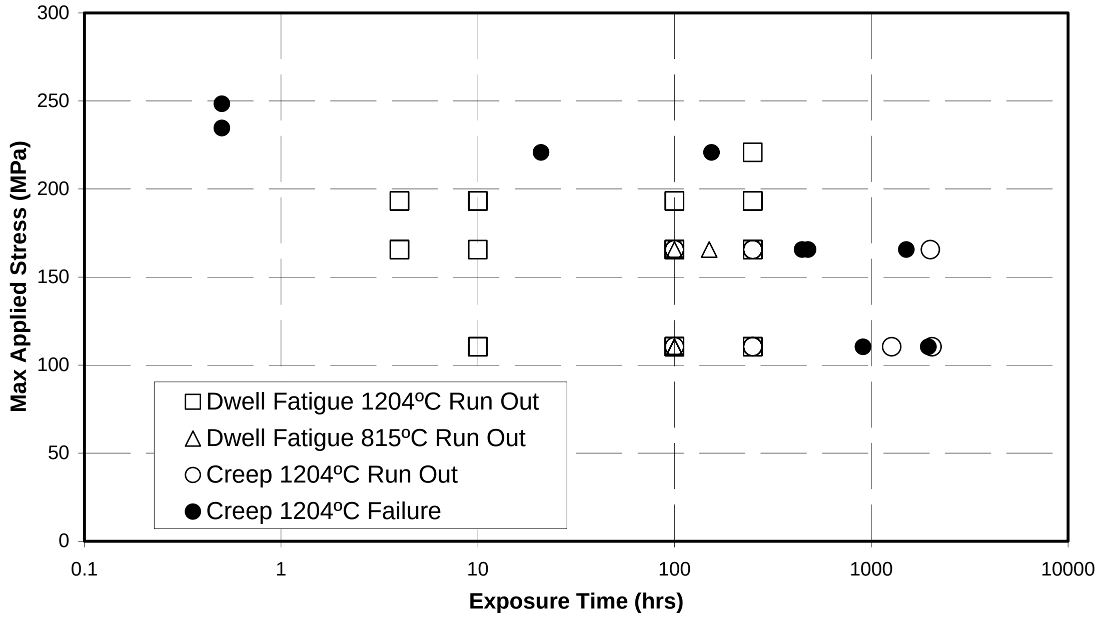
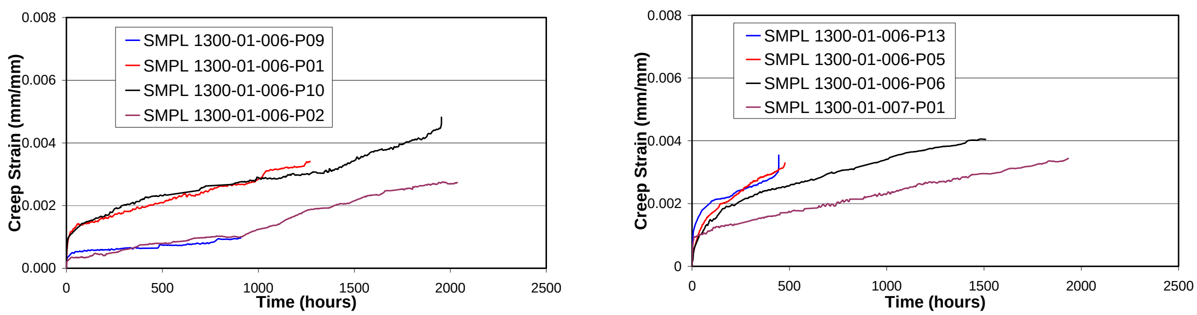


Figure 3. Creep and Dwell Fatigue Testing Summary



a) 110.4 MPa b) 165.6 MPa

Figure 4. Long-term Creep Curves

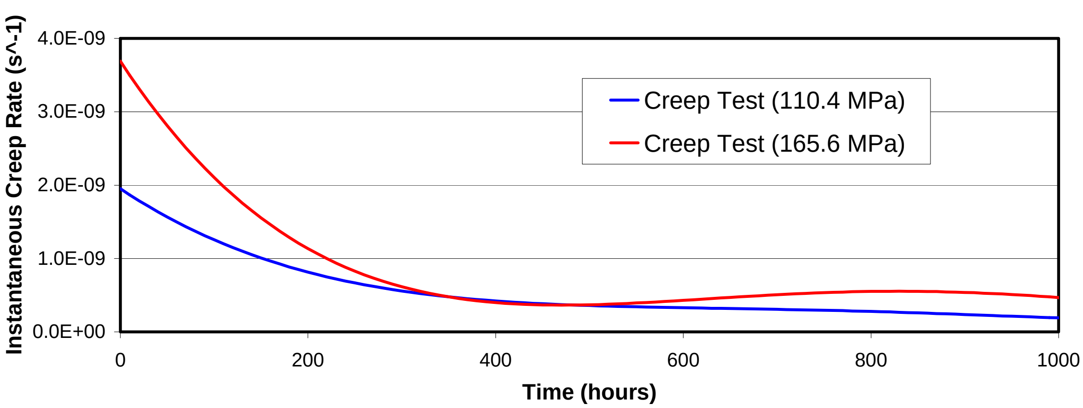


Figure 5. Instantaneous creep rate for tests done at 110.4 and 165.6 MPa

Short Term Creep Testing Analysis:

A few samples were tested in creep for durations less than 250 hours. Due to the shorter duration, an additional stress levels were added over the work shown in Figure 4. The additional stresses were 220.8 MPa, 234.6 MPa and 248.4 MPa. The two highest stress levels were very short in duration (<0.5 hours) and data collection during those tests proved difficult. Some of the longer lasting tests are shown in Figure 6 showing the resulting creep curves at 1204ºC. The curves shown for 110.4 and 165.6 MPa are consistent with the curves shown in Figure 4 in that the same strain evolution is seen (both samples were stopped at 250 hours). The strain evolution at 220.8 MPa is significantly greater and consistent with the fact that the sample failed at 153 hours. For the test at 220.8 MPa, it is not clear that a steady state region was ever achieved.

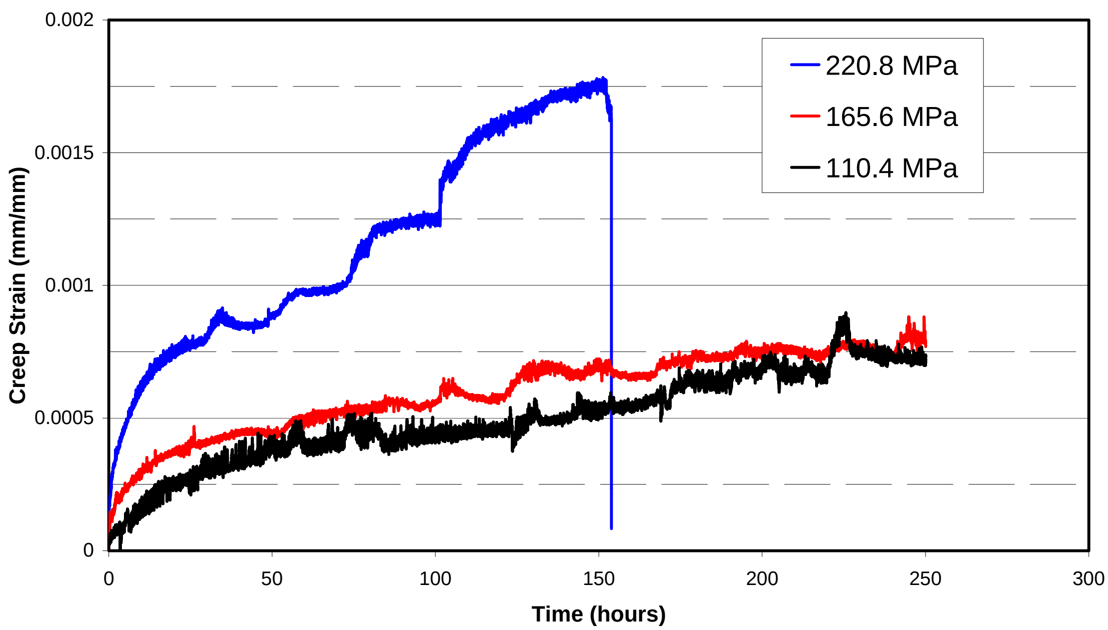


Figure 6. Short term creep curves at multiple stress levels

Dwell Fatigue Testing Analysis – Strain Evolution:

Dwell fatigue testing was done at stress levels of 110.4, 165.6, 193.2 and 220.8 MPa and no testing exceeded 250 hours. As noted earlier, the dwell duration was 2 hours before the load was cycled off and on. During the tests, total strain was recorded. For the series of tests that went to 250 hours, the total strain history is shown in Figure 7. The total strain evolved increases with increasing stress level consistent with most of the creep data shown previously. For this series of tests, the 110.4 MPa data shows less strain evolution than the creep testing effort.

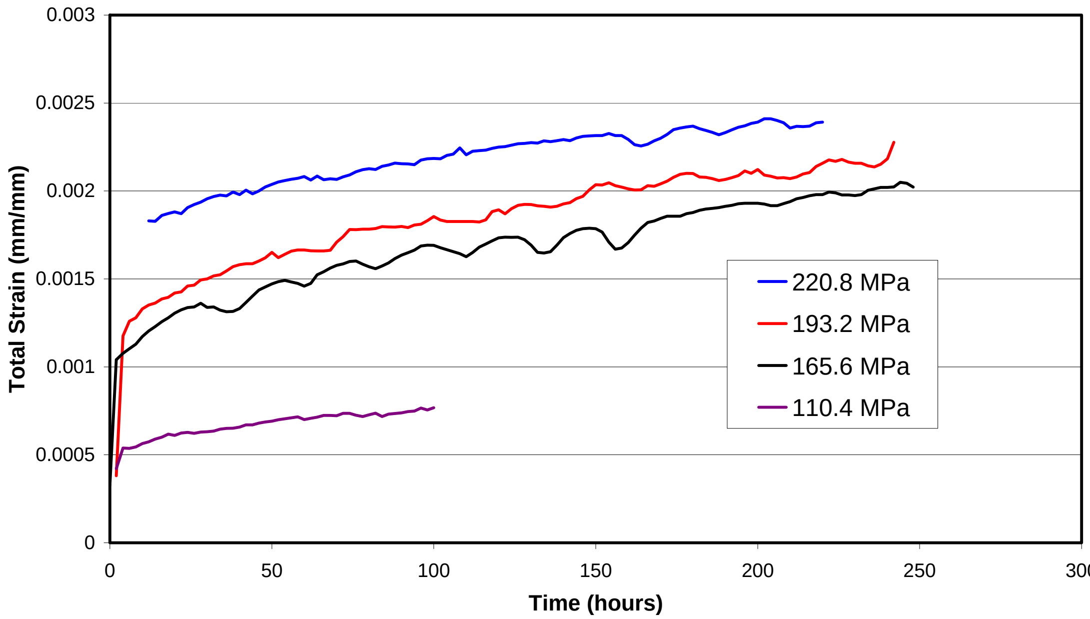


Figure 7. Strain-time history for dwell fatigue samples at various stress levels

Dwell Fatigue Testing Analysis – Modulus Evolution:

During the dwell fatigue testing, stress-strain was recorded for load-unload cycles occurred and this allowed the modulus to be determined at the start of each cycle. The change in modulus was used as an indication of damage [5]. The modulus values versus time is shown in Figure 8. As can be seen, there is early and consistent damage evolution in the high stress test results (220.8 MPa) that is not seen in the other stress levels sine they show relatively constant modulus during the test.

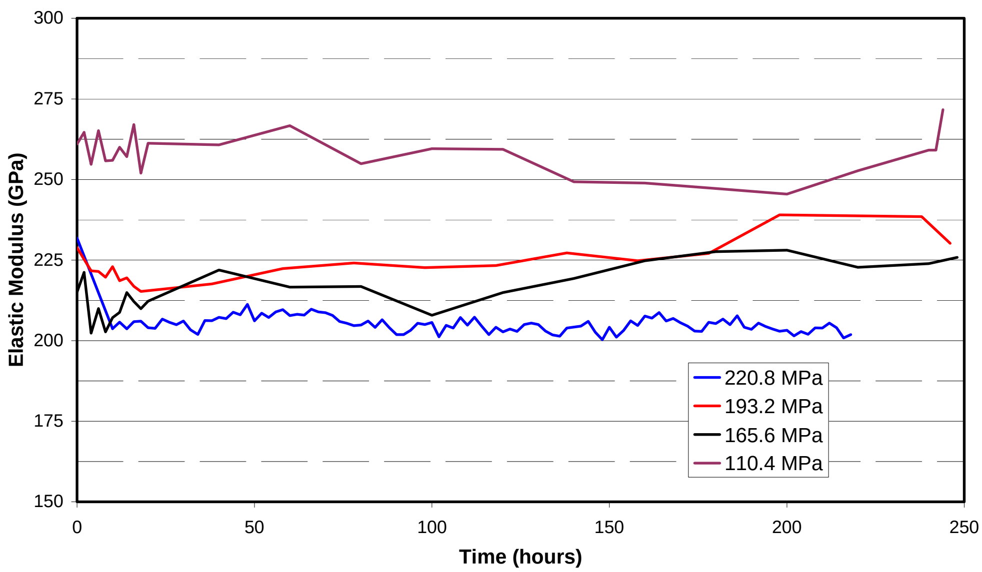


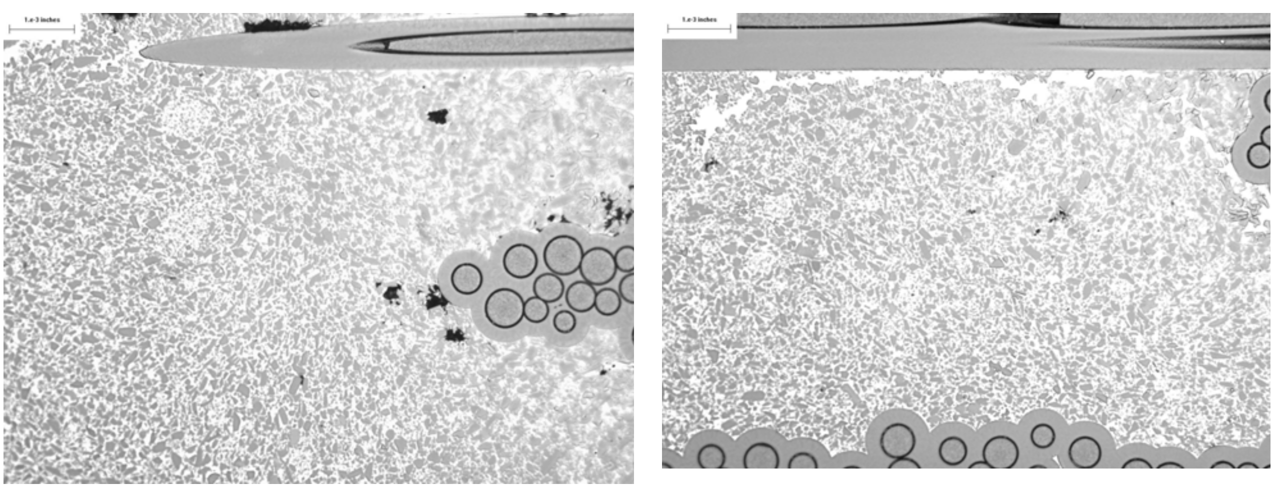
Figure 8. Modulus-time history for dwell fatigue samples at various stress levels

DISCUSSION

The work done to date shows that the MI SiC/SiC CMC is capable of sustaining loads at temperatures up to 1204ºC for long durations. As shown in Figure 3, the material did not show failures in a region of stress and time of 193.2 MPa by 250 hours. Short test durations were seen once stresses of 220.8 MPa were applied where cracking would be occurring allowing environmental attack of the fiber-interface allowing embrittlement and resulting in short term tests (note that this testing is above the proportional limit of the material as seen in Figure 2). In addition, failures at lower stresses were only seen at 450 hours or greater. At stresses of 110.4 and 165.6 MPa, durations as long as 2,000 hours were achieved. (At these times, the material still shows residual tensile capability [5].) Also, the work shows that very low creep rates are seen in this material and that the overall strain evolution is low indicating that this material will remain relatively dimensionally stable in any long-term application (see Figure 4).

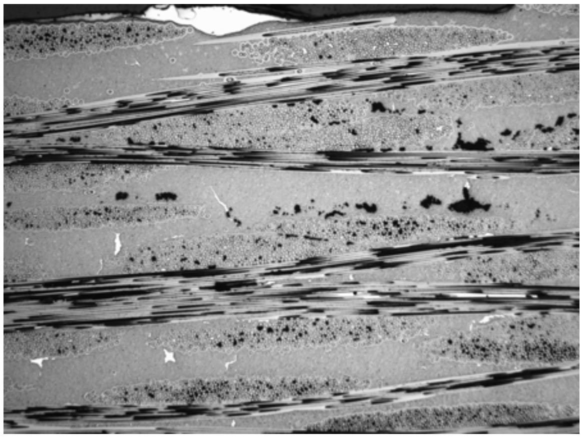
As was shown in Figures 4 and 5, the material has a relatively long primary creep region. This indicates that the damage needed to evolve in the material is time dependent and does not occur instantaneously. This is consistent, for the most part, with the modulus evolution shown in Figure 8. At the lower stress levels, the modulus values measured are remaining relatively constant showing that there is no damage evolution in the material. This was not the case for the test at 220.8 MPa where the modulus dropped after the first few cycles. This would be indicative of matrix cracking in the material at the high stress level seen (when compared to Figure 2, the stress level is above the proportional limit of the material). This is consistent with work shown by other investigators [5,7].

In order to investigate the creep behavior, some samples were sectioned and polished both near and away from the fracture location. In particular, a sample tested at 165.6 MPa and 1204ºC that showed tertiary creep was sectioned (See Figure 4b.). Cross section of this material is shown in Figure 9. This gives indication that during testing voids are forming in the material under tensile load. In addition, additional cross section work was done even closer to the fracture face as shown in Figure 10 for this sample. At the lower magnification, even additional void formation is seen. Significant voids are seen when sectioned at the failure face. There is even indications that the voids are linking up to form cracks.

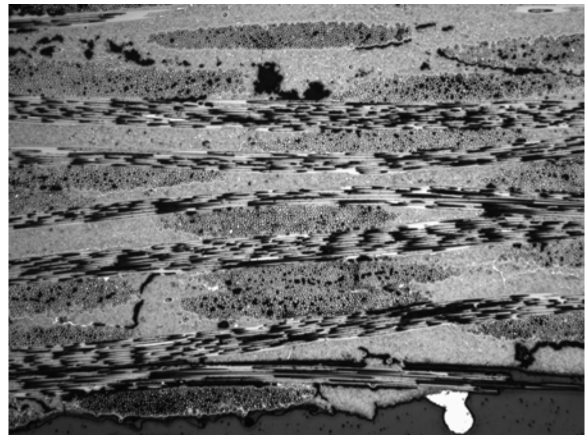


a) away from failure surface b) as manufactured

Figure 9. Optical cross section of sample tested at 165.6 MPa and 1204ºC in creep



a) away from failure face



b) at failure face

Figure 10. Additional microstructure work from sample showed in Figure 9

This effort is consistent with work done on Si/SiC where creep testing showed that voids formed at the boundaries of the Si with the SiC particulates [8-10]. As part of that work, the authors showed that the creep rate in tension was 20 times that shown in compression. As part of the confirmation of the creep testing done here, two tests were done at 165.6 MPa and 1204ºC in compression. These tests were only run for a short duration (10 hours), but no primary creep was seen. This supports the position that cavitation/void formation is occurring for the MI SiC/SiC composite at the Si-SiC particulate boundary.

CONCLUSIONS

The work has demonstrated and documented the long-term behavior of the MI SiC/SiC system. The material shows a very low creep rate and a large primary creep region. This is consistent with expectations of this CMC system. The work has shown that at the high stress of 220.8 MPa, there is clear damage measurement as seen in the modulus work. In addition, it was shown that the main damage evolution in this material was creep in the Silicon metal forming voids both in the Silicon as well as between the Silicon/SiC particulates. This is consistent with other investigators who looked at Si/SiC particulate material (same creep rates within temperature bounds and percent material) [9] and consistent with the fact that SiC typically shows creep at temperatures of 1600ºC and higher [11].

This work shows that the material is well suited for applications at the test temperatures explored here. Additionally, work should be done on the effects of holes and that is reported in the companion paper to this paper [12]. Applications that consider even longer durations than tested here should consider additional testing.

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